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IS 2600-1 (1988): Methods of chemical analysis of zinc and zinc base alloys for die castings, Part 1: Determination of copper, iron, nickel, tin and thallium by spectrophotometric method [MTD 9: Lead, Zinc, Cadmium, Tin, Antimony and their Alloys]



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IS : 2600 (Part 1) - 1988

(Reaffirmed 2003)

Indian Standard

**METHODS OF
CHEMICAL ANALYSIS OF ZINC AND ZINC
BASE ALLOYS FOR DIE CASTINGS**

**PART 1 DETERMINATION OF COPPER, IRON, NICKEL, TIN AND THALLIUM
BY SPECTROPHOTOMETRIC METHOD**

(First Revision)

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NEW DELHI 110002

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ZINC AND ZINC BASE ALLOYS FOR DIE CASTINGS

PART 1 DETERMINATION OF COPPER, IRON, NICKEL, TIN AND THALLIUM BY SPECTROPHOTOMETRIC METHOD

(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 25 July 1988, after the draft finalized by the Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 This standard was first published in 1964 and has been revised (in parts) on the basis of implementation of the standard by the manufacturers and the testing laboratories. Part 1 of the standard covers analysis of constituents by spectrophotometric methods and Part 2 covers the atomic absorption spectrophotometric

methods. With the publication of these parts covering the methods for the relevant constituents prescribed in earlier version will, therefore, be superseded. The analysis of the constituents not covered in these parts will be covered in subsequent parts.

0.3 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard (Part 1) covers spectrophotometric methods for the determination of copper, iron, nickel, tin and thallium in the range as specified in IS : 713 - 1981* and IS : 742 - 1981†.

2. SAMPLING

2.1 The sample shall be drawn and prepared in accordance with the procedure laid down in IS : 1817 - 1961‡.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, analytical grade reagents and distilled water shall be employed in the test.

4. PREPARATION OF MAIN SOLUTION

4.1 Weigh 10.00 g of sample and dissolve in 100 ml of concentrated hydrochloric acid. Evaporate to dryness. Cool, add 100 ml of water and transfer to 500 ml volumetric flask and make up.

*Specification for zinc base alloys ingots for die casting (second revision).

†Specification for zinc base alloy die castings (second revision).

‡Methods of sampling non-ferrous metals for chemical analysis.

5. DETERMINATION OF COPPER

5.1 Outline of the Method

Copper in a suitable aliquot of the main solution is complexed with sodium diethyl dithiocarbamate in ammoniacal medium is extracted with chloroform and measured at 440 nm.

5.2 Reagents

5.2.1 Ammonium Citrate Solution (Purified) — Add 210 ml of ammonium hydroxide in 50 ml of water. Add 200 ml of citric acid in small portions while stirring and cooling, and dilute to 500 ml. Transfer 100 ml of the solution to 250 ml separating funnel and make slightly ammoniacal, add 5 ml of sodium diethyl dithiocarbamate solution and mix. Add 50 ml of chloroform and shake for 1 min. Discard the organic layer.

5.2.2 EDTA Solution — 5 percent (m/v).

5.2.3 Ammonium Hydroxide — rd = 0.90.

5.2.4 Sodium Diethyl Dithiocarbamate — Dissolve 0.1 g of sodium diethyl dithiocarbamate in 90 ml water and dilute to 100 ml. Store in a amber coloured bottle (stable for a week only).

5.2.5 Chloroform

5.2.6 Standard Copper Solution (1 ml = 0.01

mg Cu) — Transfer 0.100 0 g of copper (purity 99.99 percent) to a 250 ml beaker, dissolve in 25 ml of concentrated nitric acid and add 15 ml of water. When dissolution is complete, boil to remove oxides of nitrogen. Cool and transfer to 1 litre volumetric flask and make up. Transfer 25 ml of the solution to 250 ml volumetric flask and make up.

5.3 Procedure

5.3.1 Transfer an aliquot of main solution (see 4.1) (containing 0.01 to 0.4 mg Cu) into a separating funnel. Add 10 ml of ammonium citrate solution and 20 ml of EDTA solution. Shake and adjust to pH 8.5 with ammonium hydroxide. Add 25 ml of sodium diethyl dithiocarbamate solution and mix. Add 10 ml of chloroform and shake for 2 min.

5.3.2 Allow the layers to separate and transfer the chloroform layer to 50 ml volumetric flask. Extract the aqueous layer with chloroform three times and transfer to the 50 ml volumetric flask. Make up with chloroform. Measure the absorbance of solution at 440 nm against a reagent blank.

5.3.3 Blank — Carry out a blank using same quantity of reagents as used above.

5.3.4 Calibration Curve — Take 0, 1, 2, 4, 6, 8 and 10 ml of standard copper solution (1 ml = 0.01 mg Cu) into 50 ml volumetric flasks and proceed in accordance with 5.3.1 and 5.3.2. Plot a curve of photometric readings of the calibration solution against mg of Cu/50 ml of solution.

5.3.5 Calculation — Convert the photometric readings of sample solution to mg of Cu by calibration curve. Calculate the percentage of copper as follows:

$$\text{Copper, percent} = \frac{A}{B} \times 0.1$$

where

A = mass in mg of Cu found in aliquot of sample solution, and

B = mass in g of sample represented by aliquot.

6. DETERMINATION OF IRON

6.1 Outline of the Method — Iron in aliquot of main solution is reduced and treated with *o*-phenanthroline. A red coloured complex obtained is measured at 510 nm

6.2 Reagents

6.2.1 Dilute Hydrochloric Acid — 1 : 1 (v/v)

6.2.2 Methyl Isobutyl Ketone

6.2.3 Ascorbic Acid — 0.1 percent (m/v)

6.2.4 *o*-Phenanthroline (Buffered) — Treat 1

g of reagent with 215 ml of glacial acetic acid and 265 ml of ammonia solution (rd = 0.90). Check the pH of the solution which should be 6.5 ± 0.1 .

6.2.5 Standard Iron Solution — (1 ml = 0.01 mg Fe) — Dissolve 0.500 0 g of electrolytic iron wire in 20 ml dilute hydrochloric acid (1 : 1) and evaporate on a water bath. Add 1 ml of concentrated hydrochloric acid and water. Transfer to 500 ml volumetric flask and make up. Take 10 ml of the solution and dilute to 1 litre.

6.3 Procedure

6.3.1 Take 5 ml aliquot of the main solution (see 4.1) to a separating funnel and add 20 ml dilute hydrochloric acid. Allow to stand until any turbidity disappears. Add 20 ml of methyl isobutyl ketone to the separating funnel and shake for 15 seconds. Allow the phases to separate, discard the organic phase and wash it three times with 20 ml dilute hydrochloric acid until free of copper.

6.3.2 Extract the iron from the organic layer by shaking vigorously with two successive 10 ml portions of ascorbic acid solution. Transfer the aqueous solution to 50 ml volumetric flask and add 5 ml of *o*-phenanthroline, mix and make up. Measure the absorbance of solution at 510 nm against a reagent blank.

6.3.3 Blank — Carry out a blank using same quantity of reagent as above.

6.3.4 Calibration Curve — Take 0, 2, 4, 6, 8 and 10 ml of standard iron solution (1 ml = 0.01 mg Fe) into 50 ml volumetric flasks and proceed in accordance with 6.3.1 and 6.3.2. Plot a curve of photometric readings of the solution against mg of Fe/50 ml of the solution.

6.3.5 Calculation — Convert the photometric readings of sample solution to mg of Fe by calibration curve. Calculate the percentage of iron as follows:

$$\text{Iron, percent} = \frac{A}{B} \times 0.1$$

where

A = mass in mg of iron found in aliquot of sample solution, and

B = mass in g of sample represented by aliquot taken.

7. DETERMINATION OF NICKEL

7.1 Outline of the Method — A suitable aliquot of the main solution is treated with dimethylglyoxime. Red coloured nickel soluble complex formed is measured at 445 nm.

7.2 Reagents

7.2.1 Citric Acid — Solid.

7.2.2 *Ammonium Persulphate* — Solid.

7.2.3 *Ammonium Hydroxide* — rd = 0.90.

7.2.4 *Hydroxylamine Hydrochloride* — 10 percent (w/v).

7.2.5 *Dimethylglyoxime* — 10 percent (m/v).

7.2.6 *Chloroform*

7.2.7 *Dilute Hydrochloric Acid* — 0.5 M.

7.2.8 *Bromine Water* — 10 percent.

7.2.9 *Standard Nickel Solution* — Dissolve 0.025 g of pure nickel in dilute hydrochloric acid (1:1). Add 5 ml of concentrated nitric acid and evaporate to dryness. Cool and transfer to 250 ml volumetric flask and make up (1 ml = 0.1 mg Ni). Dilute 10 ml of the solution to 100 ml (1 ml = 0.01 mg Ni).

7.3 Procedure

7.3.1 Take 25 ml aliquot of the main solution (see 4.1) in 150 ml beaker. Add 5 g of citric acid, 1 g of ammonium persulphate and excess ammonium hydroxide. Boil, cool and filter. Wash the residue three times with water and transfer the filtrate to 500 ml separating funnel. Add 1 drop of hydroxylamine hydrochloride and shake. Add 10 ml of dimethylglyoxime and shake again. Add 10 ml of ammonium hydroxide shake and allow to stand for a few minutes. Extract thrice with 10 ml chloroform. Collect the extracts in another 125 ml separating funnel.

7.3.2 Bring the nickel to ionic state by extracting thrice with 5 ml of dilute hydrochloric acid. Transfer the extracts to a 25 ml volumetric flask. Add 1 ml bromine water and 2 ml of ammonium hydroxide. Add 1 ml of dimethylglyoxime, mix and make up. Allow to stand for 5 min. Measure the absorbance of the complex at 445 nm against a reagent blank.

7.3.3 *Blank* — Carry out a blank using same quantity of all reagents as above.

7.3.4 *Calibration Curve* — Take 0, 1, 2, 3, 4 and 5 ml of standard nickel solution (1 ml = 0.01 mg Ni) into 25 ml volumetric flasks and proceed in accordance with 7.3.1 and 7.3.2. Plot a curve of photometric readings against mg of Ni/25 ml of the solution.

7.3.5 *Calculation* — Convert the photometric readings of samples solution to mg of Ni by calibration curve. Calculate the percentage of Ni as follows:

$$\text{Nickel, percent} = \frac{A}{B} \times 0.1$$

where

A = mass in mg of Ni found in aliquot of sample solution, and

B = mass in g of sample represented by the aliquot taken.

8. DETERMINATION OF TIN

8.1 *Outline of the Method* — Tin in a suitable aliquot of the main solution is treated with quercetin and the tin quercetin complex formed is extracted with methyl isobutyl ketone and measured at 440 nm.

8.2 Reagents

8.2.1 *Thiourea Solution* — 5 percent.

8.2.2 *Ascorbic Acid* — 2 percent (prepare fresh).

8.2.3 *Quercetin Solution* — Dissolve 0.5 g of quercetin in 300 ml ethanol (heat gently to dissolve). Cool and add 25 ml of concentrated hydrochloric acid. Filter off any residue and keep in a glass stoppered bottle.

8.2.4 *Methyl Isobutyl Ketone*

8.2.5 *Dilute Sulphuric Acid* — 5 percent (v/v).

8.2.6 *Standard Tin Solution* (1 ml = 0.005 mg Sn) — Dissolve 0.500 g of pure tin in 100 ml of concentrated hydrochloric acid in a 250 ml beaker. Cool and transfer to 1 litre volumetric flask and make up. Transfer 100 ml of this solution to 1 litre volumetric flask. Add 100 ml of concentrated hydrochloric acid and dilute to mark.

8.3 Procedure

8.3.1 Transfer the following reagents into a 125 ml separating funnel:

- 20 ml of thiourea,
- 5 ml of ascorbic acid, and
- 20 ml of quercetin solution and mix.

Transfer 50 ml aliquot of the main solution (see 4.1) to the above separating funnel and mix. Allow to stand for 10 to 15 min. Add 15 ml of methyl isobutyl ketone and shake for 1 min. Allow to settle the layers. Discard the aqueous phase.

8.3.2 To the organic phase, add 25 ml of sulphuric acid and shake for 30 seconds. Allow to settle and discard the aqueous phase. Measure the absorbance of the solution (organic phase) against a reagent blank at 440 nm.

8.3.3 *Blank* — Carry out a reagent blank using same quantity of reagents as above.

8.3.4 *Calibration Curve* — Take 0, 2, 4, 8, 12, 16 and 20 ml of standard tin solution (1 ml = 0.005 mg Sn) into seven 50-ml volumetric flasks and proceed in accordance with 8.3.1 and 8.3.2. Plot a curve of photometric readings against mg of Sn/50 ml of the solution.

8.3.5 *Calculation* — Convert the photometric readings of sample solution to mg of Sn by

calibration curve. Calculate the percentage of Sn as follows:

$$\text{Tin, percent} = \frac{A}{B} \times 0.1$$

where

A = mass in mg of tin found in aliquot of sample solution, and

B = mass in g of sample represented by the aliquot taken.

9. DETERMINATION OF THALLIUM

9.1 Outline of the Method — Sample is dissolved in concentrated hydrochloric acid and suitable aliquot is treated with rhodamine B reagent. The absorbance of the complex is measured at 560 nm.

9.2 Reagents

9.2.1 Concentrated Hydrochloric Acid — rd = 1.16 (conforming to IS : 265 - 1976*).

9.2.2 Hydrobromic and Bromine Water Mixture — Mix 25 ml of bromine water with 225 ml of hydrobromic acid.

9.2.3 Isopropyl Ether

9.2.4 Rhodamine B Solution — Dissolve 0.1 g of rhodamine B in 500 ml of dilute hydrochloric acid (1 N).

9.2.5 Standard Thallium Solution — Dissolve 0.1 g of pure thallium in dilute nitric acid (1:1). Add 10 ml dilute sulphuric acid (1:1). Heat to fume and cool. Dilute to 1 litre. Take 10 ml of this solution and dilute to 500 ml (1 ml = 0.002 mg thallium).

*Specification for hydrochloric acid (second revision).

9.3 Procedure

9.3.1 Dissolve 5.00 g of sample in concentrated hydrochloric acid. Heat and evaporate to dryness. Cool, add 20 ml water. Transfer to 100 ml volumetric flask. Take 25 ml aliquot of the solution and add 5 ml hydrobromic acid and bromine mixture. Heat to expel bromine. Cool and dilute to 25 ml. Transfer the solution to a separating funnel, add 10 ml of isopropyl ether and shake well. Allow the layer to separate. Drain the aqueous layer. To the ether layer, add 5 ml of rhodamine B reagent. Shake well for 1 min. Drain the reagent layer. Measure the absorbance of the complex against reagent blank at 560 nm.

9.3.2 Blank — Carry out a reagent blank using same quantity of all reagents used above.

9.3.3 Calibration Curve — Take 0, 1, 2, 3, 4 and 5 ml of standards solution of thallium (1 ml = 0.002 mg Tl) into 25 ml volumetric flasks and proceed in accordance with 9.3.1. Plot a curve of photometric readings of solution against mg of Tl/25 ml of the solution.

9.3.4 Calculation — Convert the photometric readings of sample solution to mg of Tl by calibration curve. Calculate the percentage of thallium as follows:

$$\text{Thallium, percent} = \frac{A}{B} \times 0.1$$

where

A = mass in mg of thallium in aliquot of sample solution, and

B = mass in g of sample represented by aliquot taken.

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